

D1

## PATENT ABSTRACTS OF JAPAN

(11) Publication number : 2001-199792

(43) Date of publication of application : 24.07.2001

(51) Int.Cl.

C30B 29/06  
B01D 59/00  
B01D 59/04  
B01D 59/20  
C30B 15/00  
H01L 21/02  
H01L 21/205  
H01L 21/304

(21) Application number : 2000-007224 (71) Applicant : KOMATSU ELECTRONIC  
METALS CO LTD(22) Date of filing : 14.01.2000 (72) Inventor : NAGAI SEIJI  
TOGASHI KAZUYA

## (54) SILICON INGOT AND WAFER EACH HIGH IN SILICON 28 PURITY

## (57) Abstract:

PROBLEM TO BE SOLVED: To mass-produce silicon ingot and wafer each high in silicon 28 purity without significantly changing the relevant conventional technology.

SOLUTION: The subject silicon ingot and wafer are produced by the following steps: in the production/refining process for 'monosilane gas, dichlorosilane gas, trichlorosilane gas, tetrachlorosilane gas, disilane gas, trisilane gas, or tetrasilane gas' as the upstream step for silicon single crystal production, an isotope mass separation is performed to mass-produce a relevant gas containing silicon 28 in high purity; subsequently, using the above gas, a silicon single crystal is produced using a gas feed line, gas cylinder, polycrystal oven, and single crystal (CZ, FZ) oven, each for exclusive use for the above gas, and thus the wafer as the final product is produced.

## LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of  
rejection][Kind of final disposal of application other  
than the examiner's decision of rejection or  
application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's  
decision of rejection]

[Date of requesting appeal against  
examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

## \* NOTICES \*

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## CLAIMS

## [Claim(s)]

[Claim 1] applying a centrifugal mass separation method or a fractional distillation method to "a liquid mono silane, liquid dichlorosilane, a liquid trichlorosilane, a liquid tetrapod chlorosilicane, a liquid disilane, liquid trishiran, or a liquid tetrapod silane" -- silicon 28 (silicon of the mass number 28.) the following -- being the same -- the manufacture method of high-purity-silicon 28 lump including the process which separates the fraction included in a high grade, and the process which manufactures the lump of the high purity silicon 28 of a polycrystal by using the fraction which contains this silicon 28 in a high grade

[Claim 2] The method according to claim 1 characterized by including the process which liquefies "mono-silane gas, dichlorosilane gas, trichlorosilane gas, tetrapod chlorosilicane gas, disilane gas, trishiran gas, or tetrapod silane gas" before the process which separates the fraction containing the aforementioned silicon 28.

[Claim 3] The method according to claim 2 characterized by performing liquefaction of the aforementioned mono-silane gas or trichlorosilane gas using liquid nitrogen, ethanol, an isopentane, or the Pelletier element.

[Claim 4] The process which manufactures the lump of the high purity silicon 28 of a polycrystal is the method of a publication 3 either from the claim 1 characterized by being a CVD process by using the fraction which contains the aforementioned silicon 28 in a high grade.

[Claim 5] It is the method of a publication 4 either from the claim 1 which is the mass-production-method method of the high purity silicon 28.

[Claim 6] applying a centrifugal mass separation method or a fractional distillation method to "a liquid mono silane, liquid dichlorosilane, a liquid trichlorosilane, a liquid tetrapod chlorosilicane, a liquid disilane, liquid trishiran, or a liquid tetrapod silane" -- silicon 28 (silicon of the mass number 28.) the following -- being the same -- the method of manufacturing a high grade epitaxial wafer by using the process which separates the fraction included in a high grade, and the fraction which contains this silicon 28 in a high grade for epitaxial growth

[Claim 7] How to perform high-purity-silicon 28 refining using the existing silicon refinery processing installation including the process which deposits silicon 28 preferentially using the difference of the mobility in CVD of active species including several sorts of isotopes.

[Claim 8] High-purity-silicon 28 refiner containing the CVD reactor which deposits silicon 28 preferentially using the difference of the mobility in CVD of active species including several sorts of isotopes.

[Claim 9] High-purity-silicon 28 refiner according to claim 8 which is mass-production-method equipment of the high purity silicon 28.

[Claim 10] Mass-produced silicon 28 ingot or silicon 28 wafer.

[Translation done.]

## \* NOTICES \*

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## DETAILED DESCRIPTION

## [Detailed Description of the Invention]

## [0001]

[The technical field to which invention belongs] this invention relates to the silicon ingot with high purity, the wafer, and its manufacture method of silicon of the mass number 28.

## [0002]

[Description of the Prior Art] Although the thing (4.7% of atomic percents) of 29 and the thing (3.1% of atomic percents) of 30 exist [ the mass number / the mass number other than the main things (92.2% of atomic percents) of 28 ] in the stable isotope of silicon It is reported that the kuru CHATOFU lab in Keio University and Russia succeeded in manufacture of the single crystal (following and high-purity-silicon 28 single crystal) whose purity of the silicon (following, silicon 28) of the mass number 28 is 99.92% (Nihon Keizai Shimbun, December 20, 1999). And compared with the silicon of the former [ high purity silicon / this / 28 ], 60% of thermal conductivity being good and excelling in heat dissipation nature is shown in the multiple address notice.

[0003] Here, according to the above-mentioned report, it has obtained by presenting a centrifuge method with silicon powder for high-purity-silicon 28 single crystal, and performing mass separation of a silicon isotope.

## [0004]

[Problem(s) to be Solved by the Invention] However, when powder performs mass separation of an isotope, efficiency is bad, there is a limitation also in attainment purity, and it is not suitable for mass production method. However, it is not an overstatement, although there is no technical value, if an example is taken by the present condition of silicon wafer manufacture and it cannot put on a mass-production line. For this reason, if the mass separation by the centrifugal separation of silicon powder though the property of high-purity-silicon 28 single crystal is [ how ] good must be adopted, the good property is not employed efficiently.

[0005] On the other hand, in the manufacturing process of the present silicon, since the alteration of an existing facility is remarkable and difficult, it is desirable to enable it to raise the purity of silicon 28, using the existing facility as it is.

[0006] this invention is made in view of the above technical problems, and the purpose is in enabling it to mass-produce the ingot and wafer of the high purity silicon 28, without adding a major change to a Prior art.

## [0007]

[Means for Solving the Problem] In order to solve the above technical problems, as a result of this invention persons' repeating examination wholeheartedly, by performing isotope mass separation in the stage of the mono-silane gas at the time of manufacturing polycrystal silicon, or trichlorosilane gas, possibility that mass production method will become possible is found out, and it came to complete this invention.

[0008] More specifically in this invention, the following is offered.

[0009] (1) applying a centrifugal mass separation method or a fractional distillation method to "a liquid mono silane, liquid dichlorosilane, a liquid trichlorosilane, a liquid tetrapod chlorosilicane, a liquid disilane, liquid trishiran, or a liquid tetrapod silane" -- silicon 28 (silicon of the mass number 28.) the following -- being the same -- the manufacture method of high-purity-silicon 28 lump including the process which separates the fraction included in a high grade, and the process which

manufactures the lump of the high purity silicon 28 of a polycrystal by using the fraction which contains this silicon 28 in a high grade

[0010] Here, the technique of carrying out isotope mass separation of what was liquefied in the stage of mono-silane gas or trichlorosilane gas as the technique of performing isotope mass separation in the stage of mono-silane gas or trichlorosilane gas is mentioned. The usual isotope mass separation method it is as distilling as the concrete technique of isotope mass separation \*\*\*\* [, and ] is employable. [ applying a liquid mono silane or a liquid trichlorosilane to centrifugal separation.]

[0011] (2) The method given in (1) characterized by including the process which liquefies "mono-silane gas, dichlorosilane gas, trichlorosilane gas, tetrapod chlorosilicane gas, disilane gas, trishiran gas, or tetrapod silane gas" before the process which separates the fraction containing the aforementioned silicon 28.

[0012] (3) The method given in (2) characterized by performing liquefaction of the aforementioned mono-silane gas or trichlorosilane gas using liquid nitrogen, ethanol, an isopentane, or the Pelletier element. Thus, by using liquid nitrogen etc., it can cool cheaply and becomes suitable for the mass production of a wafer etc.

[0013] (4) (1) to (3) characterized by the process which manufactures the lump of the high purity silicon 28 of a polycrystal by using the fraction which contains the aforementioned silicon 28 in a high grade being a CVD process -- either -- the method of a publication

[0014] (5) (1) to (4) which is the mass-production-method method which is the high purity silicon 28 It is the method of a publication either.

[0015] (6) applying a centrifugal mass separation method or a fractional distillation method to "a liquid mono silane, liquid dichlorosilane, a liquid trichlorosilane, a liquid tetrapod chlorosilicane, a liquid disilane, liquid trishiran, or a liquid tetrapod silane" -- silicon 28 (silicon of the mass number 28.) the following -- being the same -- the method of manufacturing a high grade epitaxial wafer by using the process which separates the fraction included in a high grade, and the fraction which contains this silicon 28 in a high grade for epitaxial growth

[0016] (7) How to perform high-purity-silicon 28 refining using the existing silicon refinery processing installation including the process which deposits silicon 28 preferentially using the difference of the mobility in CVD of active species including several sorts of isotopes.

[0017] (8) High-purity-silicon 28 refiner containing the CVD reactor which deposits silicon 28 preferentially using the difference of the mobility in CVD of active species including several sorts of isotopes.

[0018] (9) High-purity-silicon 28 refiner given in (8) which is mass-production-method equipment of the high purity silicon 28.

[0019] (10) Mass-produced silicon 28 ingot or silicon 28 wafer.

[0020] In addition, because of the mass production of silicon 28, the above this inventions can be made into the method of using a CVD reactor, and can also be caught. Moreover, on a certain side, isotope separation equipment will be used in this invention as isotope separation equipment for the single isotope chlorine manufacture for chlorosilicane manufacture supplied to a CVD reactor for the mass production of silicon 28.

[0021] Moreover, on other sides, this invention can lower the temperature of mono-silane gas or trichlorosilane gas to below each boiling point, can change it into a liquid state, can be considered as the conductive adjustment method including the process which raises the purity of silicon 28 by the centrifuge method or the fractional distillation method of silicon, and can also be caught.

Furthermore, in addition to this, according to this invention, not to mention the ability to use it again because of manufacture of the silicon wafer of the high purity silicon 28 obtained by this invention of a semiconductor device Can also produce the silicon wafer with the purity of silicon 28 higher than usual which does not go to the high purity silicon 28, and it sets to such a silicon wafer. When producing the device of atomic level or the level near it, the use which avoids the portion in which the atom of silicon 29 or silicon 30 exists, and produces the device concerned can be presented.

[0022]

[Embodiments of the Invention] In-manufacture / refining process of mono-silane gas and trichlorosilane gas which is the upstream software development of silicon-single-crystal manufacture, this invention performs isotope mass separation and mass-produces the gas concerned

which contains the silicon of the mass number 28 in a high grade.

[0023] In manufacturing high-purity-silicon 28 lump as an example First, "mono-silane gas, Dichlorosilane gas, trichlorosilane gas, tetrapod chlorosilicane gas, Disilane gas, trishiran gas, or tetrapod silane gas" is liquefied. "liquid mono silane, Liquid dichlorosilane, a liquid trichlorosilane, a liquid tetrapod chlorosilicane, A liquid disilane, liquid trishiran, or a liquid tetrapod silane" is obtained, or the commercial elegance of "a liquid mono silane, liquid dichlorosilane, a liquid trichlorosilane, a liquid tetrapod chlorosilicane, a liquid disilane, liquid trishiran, or a liquid tetrapod silane" is purchased.

[0024] And by applying to a centrifugal mass separation method to it, an isotope is separated for every weight and those high grade liquids are manufactured from the fraction of "the mono silane, the dichlorosilane, the trichlorosilane, the tetrapod chlorosilicane, the disilane, the trishiran, or the tetrapod silane" which contains silicon 28 in a high grade. Or the high grade gas of the mono silane which contains silicon 28 in a high grade, or a trichlorosilane is manufactured by using that a difference is in the ease of carrying out of evaporation by the isotope, for example, distilling fractionally commercial "liquid mono silane, liquid dichlorosilane, liquid trichlorosilane, liquid tetrapod chlorosilicane, liquid disilane, commercial liquid trishiran, or liquid tetrapod silane" (fractional distillation) (fractional distillation method).

[0025] And next, "mono silane which contains in a high grade the silicon 28 manufactured at these processes, Dichlorosilane, a trichlorosilane, a tetrapod chlorosilicane, a disilane, "The high grade liquid of trishiran or a tetrapod silane" or silicon 28 is included in a high grade. A mono silane, Dichlorosilane, a trichlorosilane, a tetrapod chlorosilicane, a disilane, The mono silane or trichlorosilane which contains silicon 28 in a high grade is obtained from the high grade gas of trishiran or a tetrapod silane." The lump of the high purity silicon 28 of a polycrystal is manufactured by presenting CVD usually performed with "the mono silane, the dichlorosilane, the trichlorosilane, the tetrapod chlorosilicane, the disilane, the trishiran, or the tetrapod silane" which contains the silicon 28 concerned in a high grade.

[0026] The boiling point of a mono silane the boiling point of -112 degrees C and dichlorosilane here 8.2 degrees C, The boiling point of a trichlorosilane the boiling point of 32.2 degrees C and a tetrapod chlorosilicane 59.0 degrees C, The boiling point of a disilane is [ the boiling point of 52.9 degrees C and a tetrapod silane of the boiling point of -14.5 degrees C and trishiran ] 107.4 degrees C. in ordinary temperature in this about a gaseous thing It can consider as a liquid easily by using liquid nitrogen (boiling point of -196 degrees C), ethanol (-114.5 degrees C of melting points), - 159.9 degrees C of isopentane melting points, the Pelletier element (a temperature control being free), etc. And as mentioned above, after changing into a liquid state, by the centrifuge method or the fractional distillation method, mass separation of an isotope will be performed and 99.9% or more of high grade gas will be manufactured.

[0027] Although it is completely the same as usual when this high grade "mono-silane gas, dichlorosilane gas, trichlorosilane gas, tetrapod chlorosilicane gas, disilane gas, trishiran gas, or tetrapod silane gas" is used, a silicon single crystal is manufactured using the gas supply line only for high grade gas, a chemical cylinder, a polycrystal furnace, and a single crystal (CZ, FZ) furnace, and even the wafer which is a final product is manufactured.

[0028] Moreover, manufacture of the epitaxial wafer which uses this high grade gas as growth gas of EPI growth, and has a high grade epilayer is also possible.

[0029] For that purpose, for example, "liquid mono silane, liquid dichlorosilane, a liquid trichlorosilane, [ whether a centrifugal mass separation method is applied to what liquefied and obtained the commercial elegance of a liquid tetrapod chlorosilicane, a liquid disilane, liquid trishiran, or a liquid tetrapod silane", or the gas corresponding to them, and ] Or by applying a fractional distillation method, the high grade gas of "the mono silane, the dichlorosilane, the trichlorosilane, the tetrapod chlorosilicane, the disilane, the trishiran, or the tetrapod silane" which contains silicon 28 in a high grade is manufactured. And a high grade epitaxial wafer is manufactured by using the high grade gas concerned as growth gas of epitaxial growth.

[0030] By the way, by choosing a suitable thing in the separation method of an isotope, it is hardly accompanied by the alteration of an existing facility, but this invention persons also offer the method of raising the purity of silicon 28, using the existing facility almost as it is.

[0031] The method of separating the single compound which contains the isotope concerned in the mobility of the chemical species in electrolysis or CVD in the single compound containing an isotope as a method for that using a difference coming out, for example can be mentioned.

[0032] For example, it sets at the process which obtains semiconductor class polycrystal silicon by returning trichlorosilane gas on the silicon mandrel heated in the CVD reactor. The trichlorosilane which unified the chlorine atom of a trichlorosilane into either chlorine 35 (chlorine atom of the mass number 35) or chlorine 37 (chlorine atom of the mass number 37) is used. It is possible by using the difference of the mobility of the chemical species (trichlorosilane) within a CVD reactor to raise the purity of silicon 28.

[0033] Since the mass number moves only a light part quickly, the trichlorosilane molecule of silicon 28 is early movable on a silicon mandrel within a CVD reactor, and can make silicon deposit on the mandrel concerned rather than the trichlorosilane molecule of other stable isotopes (silicon 29 and silicon 30), if it explains more concretely.

[0034] For this reason, by being made to carry out at low temperature if possible, and adjusting the conditions of CVD appropriately, the purity of the silicon 28 deposited on the silicon mandrel in a CVD reactor can be raised, and, finally the high purity silicon 28 can be obtained so that it may shift to the system of the reaction rate rule of CVD from thermodynamic control.

[0035] And by doing in this way, it can hardly be accompanied by the alteration of an existing facility, but the high purity silicon 28 can be obtained now, using the existing facility almost as it is. In addition, conditioning at the time of electrolyzing water and separating heavy water as conditioning of CVD, etc. is carried out to reference, and is performed. Moreover, separation of chlorine 35 and chlorine 37 can be performed by applying the usual isotope separation method.

[0036]

[Effect of the Invention] Since efficiency is high compared with powder, according to this invention, the isotope mass separation in a liquid state can mass-produce easily the silicon ingot and silicon wafer of the high purity silicon 28.

---

[Translation done.]